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# ACOUSTIC MICROSCOPY AT CRYOGENIC TEMPERATURES

## Annual Summary Report

1 July 1981 - 30 June 1982

Contract No. N00014-77-C-0412

G.L. Report No. 3481

August 1982

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)  <b>&gt; This report describes our progress on very high resolution acoustic microscopy in very low temperature liquid helium. During the past year we have recorded the first acoustic images at temperatures less than .2°K. The acoustic wavelength in helium was 2400 Å, which was the shortest ever used in acoustic imaging. After several improvements to the microscope were made, the helium acoustic wavelength was reduced to 900 Å and images recorded with sub-1000 Å resolution.</b>		

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## **A. Introduction**

Acoustic microscopy at cryogenic temperatures and research on the acoustic properties of cryogenic fluids constitute the main areas of study for this research program.

The cryogenic microscope holds promise of a new method of imaging in the sub-micron range. This range of sizes is important to a number of technologies. Materials of all kinds have to be examined with this detail. Modern integrated circuits are being scaled downward to the point where the width of the lines are less than one micron. These problems require something new in the way of imaging instruments and the cryogenic acoustic microscope holds promise of filling this need.

Associated with this is a need to extend our knowledge of the properties of cryogenic fluids such as helium. We believe that acoustic waves in the microwave region - where the sound wavelength can be less than  $1000 \text{ \AA}$  - is a unique probe for the study of these properties.

We have made substantial progress in both of these areas. The microscope now produces quality images with a resolution that enables us to image detail  $1000 \text{ \AA}$  in size. We have gained some understanding of the non-linear acoustic properties of liquid helium at these frequencies and we are planning further work in those areas for the future period.

## **B. The Helium Microscope**

During the past year of this reporting period we have made rapid progress towards our goal of ultra-high resolution imaging using acoustic waves in very low temperature liquid helium. Earlier this year, we succeeded in recording our first images at temperatures less than  $.2^{\circ}\text{K}$ ; the acoustic wavelength in the

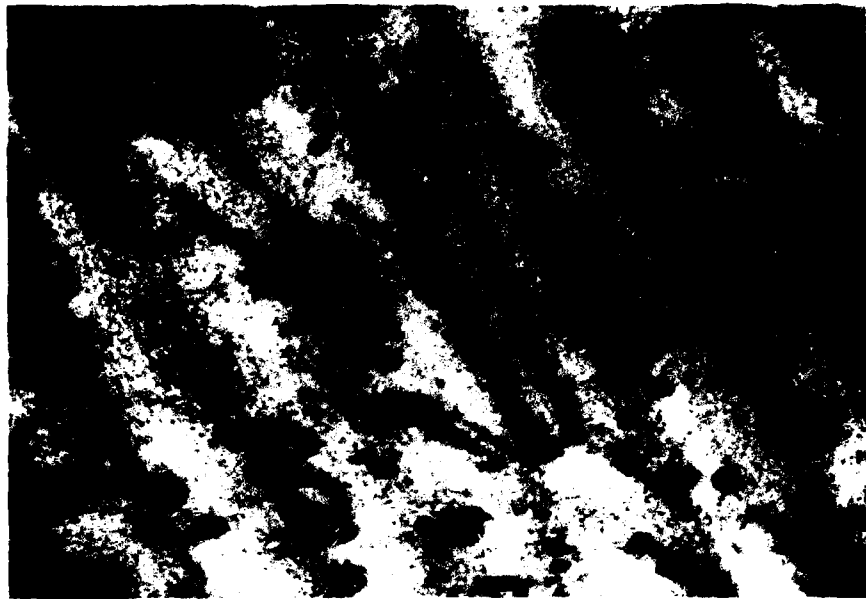
helium was  $2400 \text{ \AA}$ . At the time, this wavelength was the shortest ever used for acoustic imaging. More recently, we have significantly improved on this result by reducing the acoustic wavelength to  $900 \text{ \AA}$ . Good signal-to-noise ratio and resolution comparable to a wavelength were observed. This report describes these results in further detail.

### C. First Images and Improvements

Earlier during this reporting period we completed construction of a mechanical scanner compatible with the dilution refrigerator previously installed. The scanner was described in detail in a previous Status Report.<sup>1</sup> Early performance of the scanner was impressive and allowed us to obtain the first microscopic images in  $.1^\circ\text{K}$  superfluid helium (Fig. 1). The object is a 4 micron period grating consisting of 2 micron wide aluminum lines on a glass substrate. The grating has poor contrast in Fig. 1(a) because the aluminum lines are quite thin ( $600 \text{ \AA}$ ) and the depth of focus of the lens is large due to the small opening angle ( $15^\circ$ ). The most prominent features in the image are the structures seen with black outlines. The regions are believed to consist of a thin layer of frozen air which selectively condenses onto the aluminum grating lines. The grating is seen with greater contrast in Fig. 1(b) due to operating the microscope in a highly non-linear regime.<sup>2</sup>

In order to proceed to shorter acoustic wavelengths ( $< 1 \mu\text{m}$ ) we solved two major problems evident after these first images were taken, the inadequate focusing system and a poor signal-to-noise ratio. Mechanical focusing was performed by a micrometer attached to the room temperature end of the two meter long sample holding rod. One micron positioning accuracy was observed. However, with shorter acoustic wavelengths, the depth of focus can easily be less than .1 micron, and comparable positioning accuracy is needed. In this

→ | 4μm | ←



(a)



(b)

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FIGURE 1

regard, a piezoelectric positioner was added with  $< 10 \text{ \AA}$  accuracy for fine focusing (Fig. 2). This positioner may also be used to compensate for an unlevel sample by changing length to keep the system in focus while scanning.

The signal-to-noise ratio of our imaging at 980 MHz was less than 10 dB. At higher frequencies, we expect the signal-to-noise to be less because of the severe non-linear behavior encountered in liquid helium. Hence, improving the signal-to-noise is of primary importance in raising the operating frequency of the microscope.

The electronic noise in the microscope system is the combination of the thermal noise of source impedance and noise contributed by the receiving pre-amplifier. To improve our receiving system we have taken advantage of the cryogenic environment of the microscope and installed low-noise cooled GASFET pre-amplifiers.<sup>3</sup> Noise temperatures of approximately  $20^\circ\text{K}$  are obtained with the amplifier package cooled to liquid helium temperatures.

The source noise of our previous experiments was determined by room temperature losses in the coaxial cable and circulator which preceded the pre-amplifier. Again we have taken advantage of the cold environment around the microscope and have installed lossless superconducting coaxial lines and a directional coupler (operating at  $4.2^\circ\text{K}$ ) to replace the circulator (Fig. 3). The source noise is then negligible compared to the pre-amplifier noise.

#### D. Sub- $1000 \text{ \AA}$ Resolution Images

The source noise and pre-amplifier improvements have lowered the noise level in our system by approximately 20 dB. These improvements allowed us to advance the operating frequency of the microscope to 2.6 GHz, giving a  $900 \text{ \AA}$  acoustic wavelength in helium. The acoustic lens used in this experiment had been previously developed for use in liquid nitrogen and was easily adapted

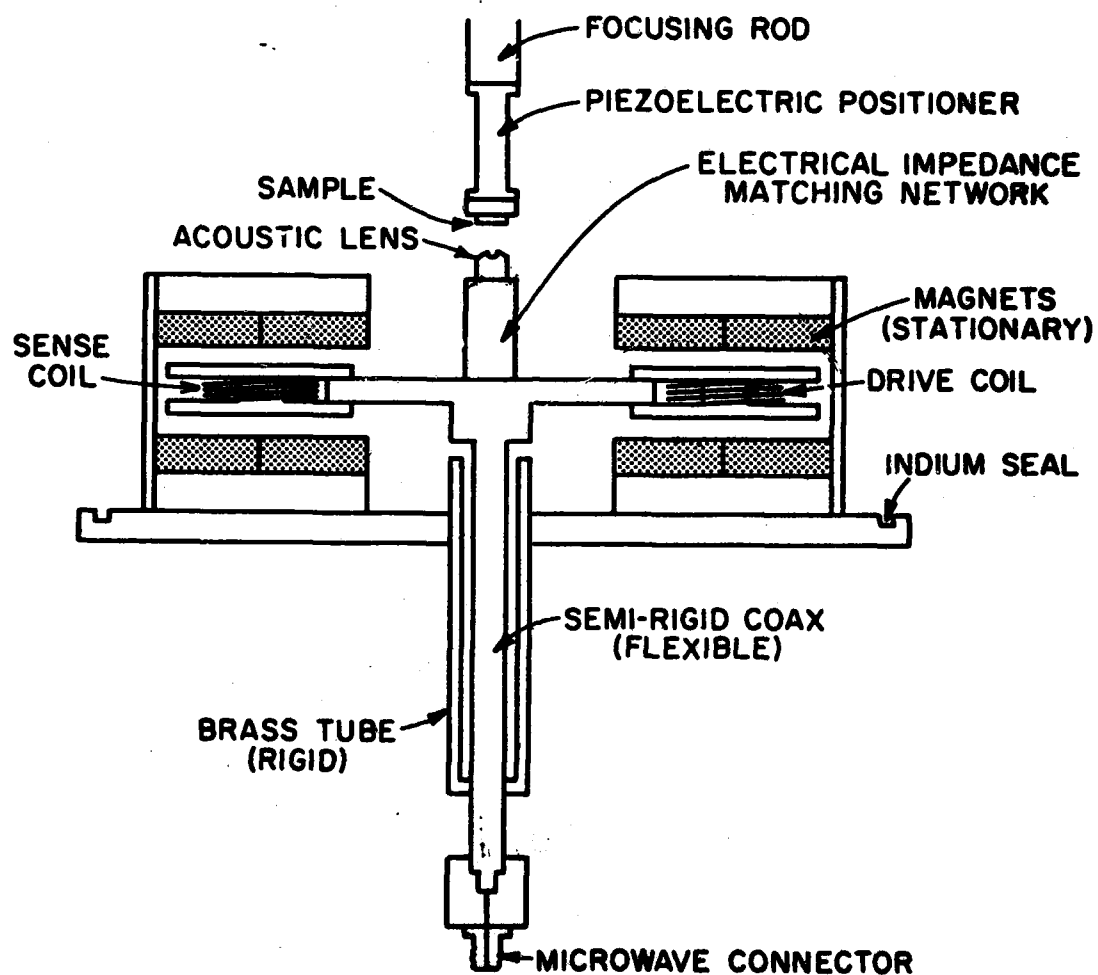
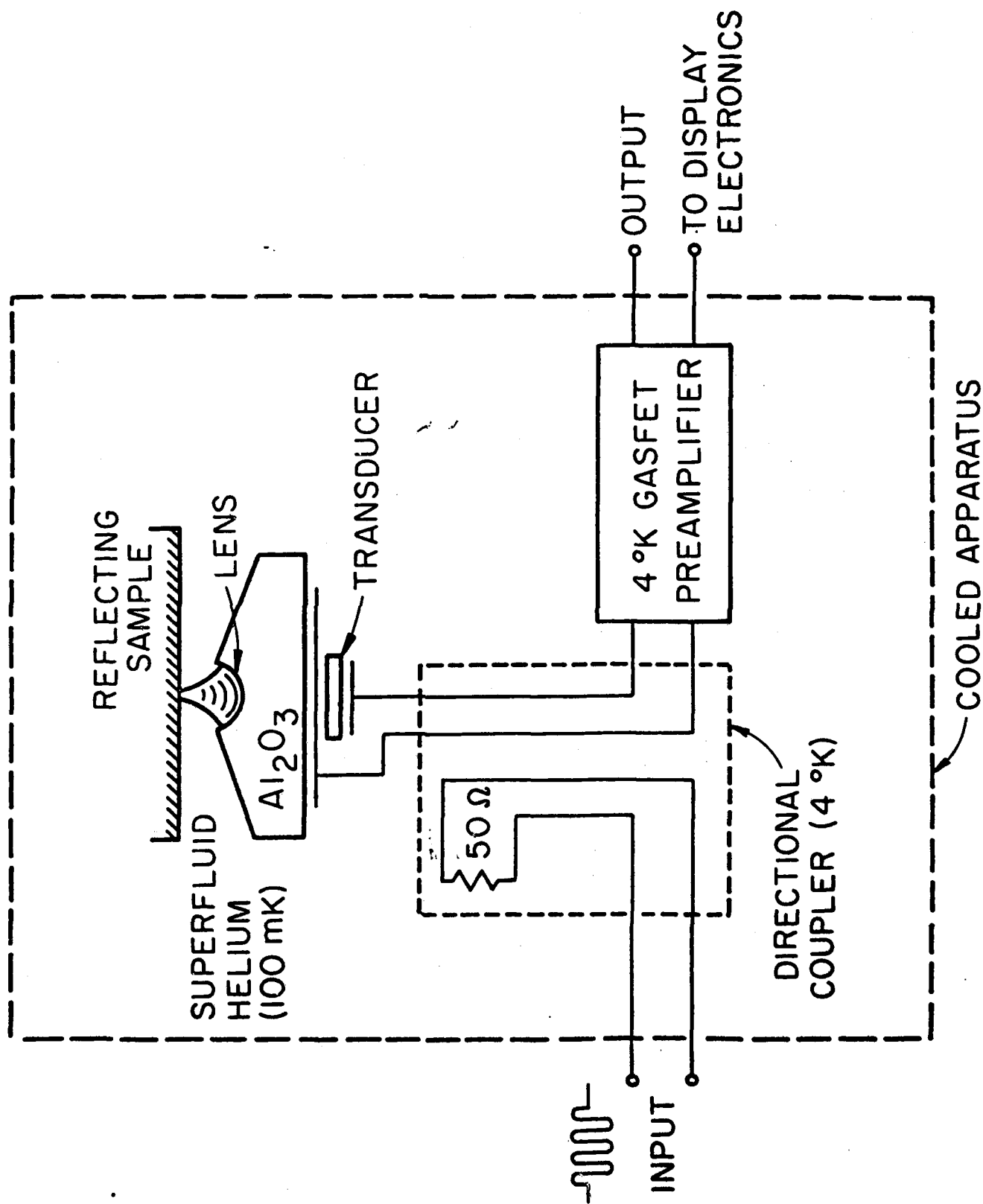


FIGURE 2





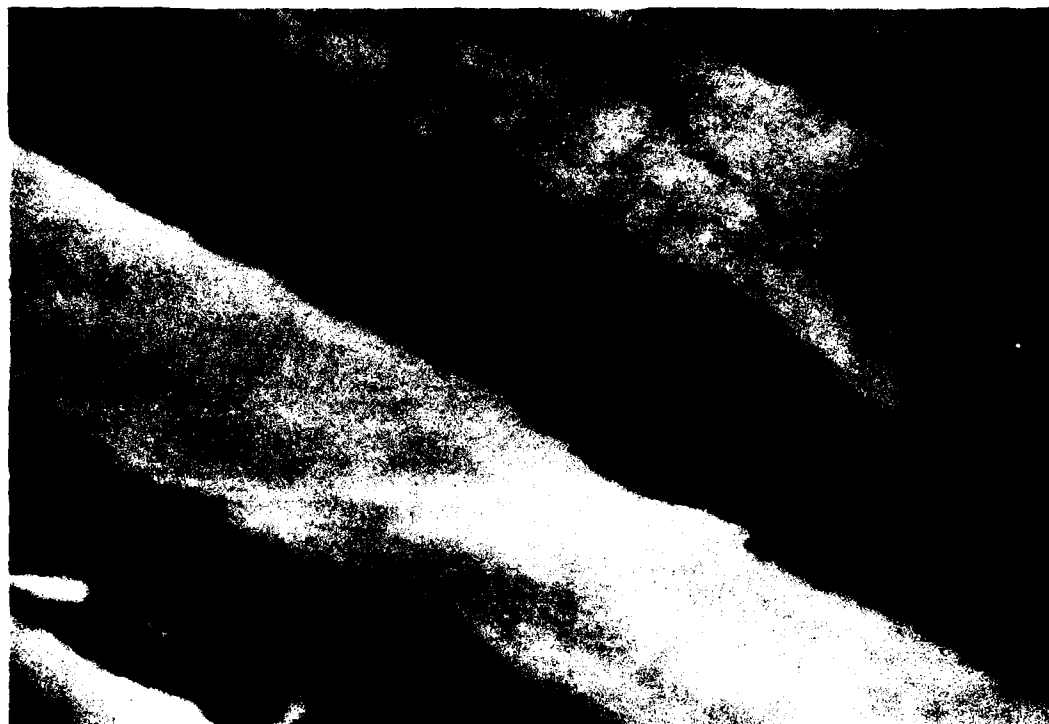
for use in helium. As the F-number of the lens is approximately .75, resolution on the order of a wavelength was expected. Figures 4 and 5 show two of the recorded images at 100 m°K and sub-1000 Å resolution is apparent. The sample is a 2 micron period grating of aluminum on glass. The aluminum steps are 600 Å high.<sup>4</sup> The signal-to-noise ratio was approximately 20 dB, a satisfactory value.

At these low temperatures in helium, acoustic attenuation in the medium is negligible for all frequencies of interest to us. As we advance in frequency toward higher resolution the constraints will be lens smoothness, scanning error and non-linear excess attenuation. The latter involves the conversion of the fundamental acoustic wave into higher harmonics and as such is an effective attenuation. This effect becomes more pronounced at higher frequencies, and will require further improvements in signal-to-noise. Several options are under consideration. Further resolution improvements are expected as well as more non-linear acoustics research and imaging applications.

One exciting application for the low temperature acoustic microscope is the possibility of imaging vortices in type II superconductors. A source of contrast for the vortices is the acoustic attenuation difference between normal and superconducting regions. We have recently measured an observable change in attenuation in a thin film (3 microns) of superconducting tin (type I) when driven normal by a magnetic field. The acoustic frequency was 2.6 GHz and the temperature was held constant at 1.5°K. Details of imaging vortices in superconductors are under consideration.

# IMAGING IN HELIUM

$T=0.2\text{ }^{\circ}\text{K}$ ,  $F=2.6\text{ GHz}$ ,  $\lambda=900\text{ \AA}$



$1\text{ }\mu\text{m}$

FIGURE 4

# IMAGING IN HELIUM

$T=0.2\text{ }^{\circ}\text{K}$ ,  $F=2.6\text{ GHz}$ ,  $\lambda=900\text{ \AA}$



1  $\mu\text{m}$

FIGURE 5

## REFERENCES

1. Status Report, 1 July 1981 - 1 January 1982, G.L. Report No. 3388.
2. Ibid.
3. S. Weinreb, IEEE Transactions on Microwave Theory and Techniques, MTT-28, No. 10, 1041 (1980).
4. Our thanks to W. R. Shreve (Hewlett-Packard) for mechanical measurements.

## Miscellaneous Activities during this Report Period

### Publications

J. Heiserman, "Acoustic Measurements in Superfluid Helium", Section 8 in Methods of Experimental Physics, Vol. 19 (Ultrasonics) P. Edmonds, ed. (Academic Press, 1981) pp. 413-453.

J. Heiserman, "Thermal Grounding of a Transmission Line in a Dilution Refrigerator", Cryogenics, 243-244 (May 1982).

### In press:

J. Heiserman, "Cryogenic Acoustic Microscopy: the Search for Ultrahigh Resolution using Cryogenic Liquids". To be published in Physica (North-Holland Publishing Company).

### Meetings and Invited Talks

J. Heiserman - Invited paper, Sixteenth International Conference on Low Temperature Physics (LT-16), August 19-26, 1981, University of California at Los Angeles, California. Paper entitled "Cryogenic Acoustic Microscopy: the Search for Ultrahigh Resolution using Cryogenic Liquids".

D. Rugar - 1981 IEEE Ultrasonics Symposium, October 14-16, 1981, Chicago, Illinois. Abstract entitled "Resolution Improvement in the Acoustic Microscope using High Intensity Focused Beams".

C. F. Quate - Invited talk, Surface Science Symposium with the People's Republic of China, sponsored by Xerox Palo Alto Research Center, California, October 20, 1981. Talk entitled "Acoustic and Photoacoustic Microscopy in the Study of the Elastic Properties of Surfaces".

C. F. Quate - Invited talk, Naval Postgraduate School, Monterey, California, 23rd October, 1981. Talk entitled "Acoustic Microscopy".

D. Rugar - 103rd Meeting of the Acoustical Society of America, April 26-30, 1982, Chicago, Illinois. Abstract entitled "Theory of Resolution Improvement in a

Focused Acoustic Imaging System using High Intensities", J. Acoust. Soc. Am. 71, Suppl. 1, S30.

C. F. Quate - Invited talk, Oxford University, Department of Electrical Science, Oxford, England, April 29, 1982. Talk entitled "Imaging with Scanning and Acoustics".

C. F. Quate - Invited Colloquium, University of California, Davis, Department of Physics, June 1, 1982. Talk entitled "Acoustic Imaging and Microscopy".

#### Awards

D. Rugar	-	1981-82 F. V. Hunt Fellowship in Acoustics, Acoustical Society of America.
C. F. Quate	-	1981 IEEE Morris N. Liebmann Award.
C. F. Quate	-	1982 Rank Prize for Opto-Electronics.

## FIGURE CAPTIONS

1. Images taken in liquid at approximately  $.1^{\circ}\text{K}$ . The object is a  $4\text{ }\mu\text{m}$  period grating consisting of aluminum lines on glass.
2. Acoustic Microscope Scanner.
3. Configuration for cryogenic acoustic microscopy.
4. Sub- $1000\text{ }\overset{\circ}{\text{A}}$  acoustic image.
5. Sub- $1000\text{ }\overset{\circ}{\text{A}}$  acoustic image.



